State of W		64-38	3-488	81	TOXIC AND Form 4400-91	HAZARDOUS SP	PILL REPORT Rev. 6-86
		State Div. Emer U.S. Nat'l. Respondent Chemtrec/Pestic	onse Center	(608) 266-3232 (800) 424-8802 (800) 424-9300	S	Spill ID Number 930830-04 Y Y M M D D 0-	4
Date of Incident	Day of Week	Time of Incident	A.M.	Reported By (N	(ame)	Telephone Nur	
08-30-93	man	3.50	P.M.	Leg 11	100ne		735 7033
Date Reported	Day of Week	Time Reported	A.M.	Agency or Firm			Div. Emergen.
Substance Involved	man	4.50	P.M.	Person of Firm	ty Offen	1 0000	Yes No
Methyl A	m-10	Quantity Units			ice HY (	Hem "	credi percio
Substance Involved	MARKE	Quantity	Units	Contact Name	ice Hy!	Telephone Nur	mher
Substance involved		quantity	Omis	/ m	Dre	(7/5)=	725-5033
Physical Characteristic	cs		10	AVVI	et or Route	1	27000
		0-1-	-	2	STANTON	ST	
Solid	Liquid	Color		City, State, Zip		114	-10/4 14 - 11 - 1
Semisolid	Gas	Odor M	A IND Leave	mani		5 54/43	
Cause of Incident Rupture Exact Location Descri	ption (intersect	ion, mileage, etc.)	e an vess	Taken  Containmen	No Notif	fication VIn	vestigate
				Cleanup; Mo		ONE.	
		, TN	, R	Amount Re		blew to N.W.	ma dances
DNR Dist DNR Area	Groundwaters			Monitor _		DIEW 40 V. W.	PATO
LMO MAR	☐ Yes		otential	Contractor	Hired; Name		
Surface Waters Affect		Name of Surface	Water	U Other Actio	on		
Yes No	☐ Potential			Spill Location			
Date District Notified	Day of Week	Time District No		Industrial I	Facility/Paper Mi	ll/Chem. Co.	
Ivodilicu			A.M.	Gas/Service	e Station/Garage,	Auto Dealer, Repair	Shop
District Person Notifie	d	Telephone Numb	☐ P.M.	Ag Coop/Fa	acility/Cheese Fac	ctory/Creamery	
District Person Notifie	eu .	relephone Numb	ier	Other Small Business (bank, grocery, insurance co., etc.)			
Date Investigated	Day of Week	Time Investigate	ed .	Public Prop	perty (city, county	y, state, church, school	ol, etc.)
8-3093	1		A.M.	Utility Co., Power Generating/Transfer Facility			
MON MON 9:40 1 P.M.			Private Property (home/farm)				
Person Investigating	1	Telephone Numb	er			rm, Oil Jobber/Wholes	aler
Steven 1	Refe	1715 173	20101				,4101
Action Taken By DNF	2			Transportation Accident, Fuel Supply Tank Spill			
No Action	1	Super	rvise/Conduct		Transportation Accident, Load Spill		
☐ Taken	Investiga	1	nup/	Construction, Excavation, Wrecking, Quarry, Mine			
Spiller Required To METW (MOONE @ MS			Spilled Substance Destination				
☐ Take Action; Type	Off	Tick	-				
Contractor Hired	ac .			Air Soil RECEIVED			
□ By DNR; Name							
Amount Recovered			Groundwater SED 2 0				
29.29 Enforcemen	t			Surface Water			
Other Agencies on Scene				Storm Sew	EMERIC	8.00	
Mal Plans Tina				Sanitary Sewer  Sanitary Sewer  Contained/Recovered  Other			
Local Menomoney Collective							
manuette Fine Dept. 19/169				Other Person Filing This Report (print name)			
State		Close	a un	Person Filing T	his Report (print	name)	
N. R.C. Wash				Signature		l D	ate Signed
Federal // 1		TP.	MARGICO	Signature		D	ate Signed
Additional Comments:	asc bl	ew@50,	PSI 1	when the	N 90 p.si.	AS SUPPOS	ed to
Initial 1	reports	500/bs 0	of prea	luct. Now	s probes	by less the	20 /00
Report	200	his incl	bent,	to be til	led with	GOUT CE	generes
19ter	113 Ce	Dek. Aus	Rept	-nom 6	-135pi	11-to be so	HAVICA



## SPECIALTY CHEM<sup>TM</sup> PRODUCTS CORPORATION

MEMBER, CHEMDESIGN® GROUP

TWO STANTON STREET, MARINETTE, WI 54143
(715) 735-9033
FAX NO. (715) 735-5304

## QUALITY: THE FIRST TIME, EVERY TIME

September 16, 1993

Warden Stephen Daye Wisconsin Department of Natural Resources P. O. Box 16 Marinette, WI 54143

Dear Warden Daye:

This is the follow-up written report on the accidental release of methylamine (monomethylamine) which had been reported to the DNR on August 30, 1993.

Incident Description: At approximately 0345 hours on August 30, 1993, a 4 inch graphite burst disk rated at 90 pounds per square inch ruptured at the normal operating pressure of about 50 pounds per square inch on reaction vessel 5202. Approximately 655 gallons of reaction mixture was released, but most was contained by the building 52 blow down tank. The vent to the methylamine absorber was quickly opened and full cooling was applied to vessel 5202, significantly reducing the amount of material released to the blow down tank and to the environment. Based on material balance, approximately 56 gallons of reaction mixture was released, of which, 347 pounds was methylamine.

Reaction vessel 5202 (capacity 3850 gallons) was in use to produce MAADMA. Methylamine is one of the reactants. The reaction was at the step in the reaction sequence where all reactants have been charged and the mixture is being heated to 205° F where it would be held to allow for completion of the reaction. The normal operating pressure during this operation is about 50 to 55 psig. The heat-up was about 2 hours into the normal 3 hour heating period and the temperature was 160° F. At this point, the vessel contained a total of 3638 gallons, leaving a head space of approximately 16 inches to the rupture disk.

There was no evidence of any unusual activity until the disk failed.

Letter Warden Stephen Daye Wisconsin DNR September 16, 1993 Page 3

We are also reviewing the system used for the containment of the material leaving the vessels to determine if there is a means to contain the methylamine in case of another disk rupture.

Sincerely,

Leonard O. Moore, Ph.D.

V.P., Environmental Affairs

Joseph G. Acker

President, General Manager

LOM: 1m

cc: R. Brooks, R. Eby, R. Brinkley.

E. Sedor, L. White, G. Kandow, P. Everhart. M. Ford, Chief, Marinette Fire Department.

R. Falkenberg, Chief, Menominee Fire Department.

Letter Warden Stephen Daye Wisconsin DNR September 16, 1993 Page 2

All appropriate agencies were notified:
 Marinette Fire Department
 Marinette Police Department
 Menominee Police Department
 Director of Emergency Government
 Ansul Security
 Wisconsin State Emergency Response
 Department of Natural Resources
 National Response Center

The Menominee police patrolled the southern part of Menominee, but were unable to detect any amine odor.

No evacuations were required, nor were there any complaints received from residents in either Marinette or Menominee. Three employees of Ansul complained of respiratory irritation, but, according to Carl Servatius, Director of Safety, of Ansul, none required treatment.

Mitigation and Clean-up: There was no material to be cleaned up, since the release from the vessels was specifically into the air.

The "MAADMA Post-Release Start-Up Checklist" was completed to assure that the blow down tank was emptied and cleaned and all systems were functional before proceeding with the production.

<u>Prevention of Future Incidents:</u> Several courses of action are being pursued to enable us to prevent the occurrence of future incidents of this type.

The manufacturer of the burst disk was contacted to determine why the disk burst 45 % below its stated limit and to confirm that the disk was being used at conditions and with chemicals that would be appropriate. We have also asked the disk manufacturer to estimate the useful life expectancy of the disk in this service.

The possible use of a pressure relief valve in conjunction with a rupture disk is being considered. This would minimize the amount released if the rupture disk fails.

## SUBSTANCE SPILL/RELEASE ALERT FORM

## Spill No./Notification **Date and Military Time**

Information should be reported to: Department of Military Affairs Wisconsin Division of Emergency Government 4802 Sheboygan Ave., Room 99A, P.O.Box 7865 Madison, Wisconsin 53707-7865 (608) 266-3232

Date and Military Time of Incident

93*08<u>3</u>0* <u>03</u>50

I -	EAY /6	08) 266-1569	Courte AA	PRINETTE			
			County:/ 5/7	MUDENC			
REPORTING INFORMATION Person/Firm							
Reported by: DR.	EUNARD MOORE	Responsible: S/E	CIALTY CHEN	4. TRODUCT			
(name) 2-STAN	1701) 87	Address 2	574117011				
Address & - O (F)	Till	Address	21/10/00				
City MARINETTE	State Wis Zip	7	ZIE State Wis	_ Zip			
Telephone 715-735-9035 Telephone 115-135-9035							
Spill Contact Person/Firm:	<u> </u>	Address					
(if different from above)							
Telephone		City	State	Zip			
	SUBSTANC	E INFORMATION					
Name of Substance/ Quantity Involved:							
	[ ] EHS Chemical	[ ] CERCLA Chemical [	1 Unknown				
CAS # Placard #	Quantity Solid Powde	Radi r Liquid Vapor Gas Activ		Odor			
		. /					
	CITE IN	ECRMATION					
SITE INFORMATION  Exact Location of Spill/Release (include Street Address, County, City, Village, Town, Highway, Inter-							
section, Mileage, etc.):	Telease (Ilicidde Stieet Ac	adiess, County, City, VIII	age, rown, mgnwa	iy, iiiter			
	INTON STA	1110		1 in()			
× . // /	1010N 211	EEI - / MI	NELLE	174.			
Source	of Spill	Spill	Destination				
[] Transportation [4 Ma	anufacture [] Agriculture		. /				
	and doctors to a right route and	[ 1 Soil	VAir	[ ] Water			
[] Public Property [] Co		[] Soil .	CAIr	[] Water			
[ ] Public Property [ ] Co	onstruction [] Business	[] Storm [] Sa		d/ [] Other			
X-	onstruction [] Business	[]Storm []Sa Sewer S	nitary [ ] Containe	d/ [] Other ed			
[] Private Property [] (	onstruction [] Business	[] Storm [] Sa Sewer S [] Surface Water	nitary [] Containe	d/ [] Other ed			
[ ] Private Property [ ] C  Weather Cond Wind Speed	onstruction [] Business	[]Storm []Sa Sewer S	nitary [ ] Containe	d/ [] Other ed			
[] Private Property [] C  Weather Cond  Wind Speed and Direction	onstruction [] Business	[] Storm [] Sa Sewer S [] Surface Water Name:	nitary [ ] Containe	d/ [] Other ed Potential			
[] Private Property [] C  Weather Cond  Wind Speed and Direction	Onstruction [] Business Other ditions [] Snow [] Fog	[] Storm [] Sa Sewer S [] Surface Water Name: [] Ground Water Distance to nearest d	nitary [ ] Containe ewer Recover [ ] Yes [ ] No [ ]	d/ [] Other ed Potential			
Weather Cond Wind Speed and Direction [ ] Rain [ ] Sleet [ ] Other:	onstruction [] Business Other ditions [] Snow [] Fog  PUBLIC HEALT	[] Storm [] Sa Sewer S [] Surface Water Name: [] Ground Water Distance to nearest d	nitary [ ] Containe ewer Recover [ ] Yes [ ] No [ ]	d/ [] Other ed Potential			
Weather Cond Wind Speed and Direction [] Rain [] Sleet [] Other:  Recommended safety p	onstruction [] Business Other  ditions  [] Snow [] Fog  PUBLIC HEALT  precautions (known acute/o	[] Storm [] Sa Sewer S [] Surface Water Name: [] Ground Water Distance to nearest d	nitary [ ] Containe ewer Recover [ ] Yes [ ] No [ ]	d/ [] Other ed Potential			
Weather Cond Wind Speed and Direction [] Rain [] Sleet [] Other:  Recommended safety p	onstruction [] Business Other ditions [] Snow [] Fog  PUBLIC HEALT	[] Storm [] Sa Sewer S [] Surface Water Name: [] Ground Water Distance to nearest d	nitary [ ] Containe ewer Recover [ ] Yes [ ] No [ ]	d/ [] Other ed Potential			
Weather Cond Wind Speed and Direction [] Rain [] Sleet [] Other:  Recommended safety p	onstruction [] Business Other  ditions  [] Snow [] Fog  PUBLIC HEALT  precautions (known acute/o	[] Storm [] Sa Sewer S [] Surface Water Name: [] Ground Water Distance to nearest d	nitary [ ] Containe ewer Recover [ ] Yes [ ] No [ ]	d/ [] Other ed Potential			
[] Private Property [] Condition   Weather Condition   Wind Speed and Direction   [] Rain [] Sleet   [] Other:	Onstruction [] Business Other  ditions  [] Snow [] Fog  PUBLIC HEALT  Precautions (known acute/o	[] Storm [] Sa Sewer S  [] Surface Water  Name:  [] Ground Water  Distance to nearest di  TH INFORMATION  chronic health risks):	nitary [] Contained ewer Recoverd [] Yes [] No [] The contained ewer with the contained ewer well:	d/ [] Other ed Potential			
[] Private Property [] Compared to the second secon	onstruction [] Business Other  ditions  [] Snow [] Fog  PUBLIC HEALT  Precautions (known acute/o	[] Storm [] Sa Sewer S  [] Surface Water  Name:  [] Ground Water  Distance to nearest di  TH INFORMATION  chronic health risks):	nitary [ ] Containe ewer Recover [ ] Yes [ ] No [ ]	d/ [] Other ed Potential			
Weather Cond Wind Speed and Direction [] Rain [] Sleet [] Other:  Recommended safety p  Injuries [ Fatalities []	Onstruction [] Business Other  ditions  [] Snow [] Fog  PUBLIC HEALT  Precautions (known acute/o	[] Storm [] Sa Sewer S  [] Surface Water  Name:  [] Ground Water  Distance to nearest di  TH INFORMATION  chronic health risks):	nitary [] Contained ewer Recoverd [] Yes [] No [] The contained ewer with the contained ewer well:	d/ [] Other ed Potential			
[] Private Property [] Compared to the second secon	onstruction [] Business Other  ditions  [] Snow [] Fog  PUBLIC HEALT  precautions (known acute/off)  O Unknown Yes Number  ] [] [] []	[] Storm [] Sa Sewer S  [] Surface Water  Name:  [] Ground Water  Distance to nearest di  TH INFORMATION  chronic health risks):	nitary [] Contained ewer Recoverd [] Yes [] No [] The contained ewer with the contained ewer well:	d/ [] Other ed Potential			
Weather Cond Wind Speed and Direction  [] Rain [] Sleet  [] Other:  Recommended safety p  Injuries [ Fatalities [ Facility Evacuated [ Public Evacuated [ Shelter facility:	onstruction [] Business Other  ditions  [] Snow [] Fog  PUBLIC HEALT  precautions (known acute/off)  O Unknown Yes Number  ] [] [] []	[] Storm [] Sa Sewer S  [] Surface Water  Name:  [] Ground Water  Distance to nearest d  TH INFORMATION  chronic health risks):	Contained Recovery Re	d/ [] Other ed  Potential  ] Potential  feet			
Weather Cond Wind Speed and Direction [] Rain [] Sleet [] Other:  Recommended safety p  Injuries [ Fatalities [ Facility Evacuated [ Public Evacuated [ Shelter facility: Hospital [	onstruction [] Business Other  ditions  [] Snow [] Fog  PUBLIC HEALT  precautions (known acute/off)  O Unknown Yes Number  ] [] [] []	[] Storm [] Sa Sewer S  [] Surface Water  Name:  [] Ground Water  Distance to nearest d  TH INFORMATION  chronic health risks):  Shelter Los	Contained Recovery Re	d/ [] Other ed  Potential  ] Potential  feet			
Weather Cond Wind Speed and Direction  [] Rain [] Sleet  [] Other:  Recommended safety p  Injuries [ Fatalities [ Facility Evacuated [ Public Evacuated [ Shelter facility:	onstruction [] Business Other  ditions  [] Snow [] Fog  PUBLIC HEALT  precautions (known acute/off)  O Unknown Yes Number  ] [] [] []	[] Storm [] Sa Sewer S  [] Surface Water  Name:  [] Ground Water  Distance to nearest d  TH INFORMATION  chronic health risks):  Shelter Los	CED Contained Printing Water Well:	d/ [] Other ed  Potential  ] Potential  feet			

	RESPONSE INFORMATION					
Local Responder on Scene (Give name)  [] Fire MARINETTE						
[] Law Enforcement						
[] DNR KANDY STAK	Title CHIEF					
[ ] County EG						
[] EMS	Telephone 7/5-735-3342					
[ ] Other:	[ ] No further follow-up or state					
assistance required  List of contacts made by responsible party/firm or individual reporting incident:						
Agency	Telephone Number Contact Name					
[ ] Dist. Dept. of Natural Resources (Di						
[ ] Loc. Emer. Planning Committee (LE						
[ ] Local Public Health						
	1,000,404,0000					
[] CHEMTREC	1-800-424-9300					
Narrative: Cause of incident/Actions being taken/Other information:						
	SK FAILED					
- norrone a,						
	DUTY OFFICER ACCUSAGE					
	DUTY OFFICER CONTACTS  Telephone Number Contact Name					
Agency [ ] DNR (days)	Telephone Number Contact Name(608) 266-2141					
[] DNR (Duty Officer pager)	_(000) 200-2141					
[] DOT/State Patrol						
[] DH&SS						
[] DILHR						
[] DATCP						
[] NRC	_1-800-424-8802					
[] Area Director						
[ ] DEG, Dir. Bureau of Field						
Services and Disaster Resources	OLLOW-UP (to include date and time)					
	71-1-10-10					
(10000)	DENT IN TO THE ATMOST HERE.					
	ACENOV DEFINITIONS					
DEG Div. of Emerg Government	AGENCY DEFINITIONS  NRC Nat'l Response Center (Federal Reporting					
CHEMTREC Chemical Transportation Emerg. Center Environmental Protection Agency US Coast Guard)  DNR Department of Natural Resources DILHR Dept. of Industry, Labor and Human Relations						
DOT/State Patrol Dept. of Transportation	DATCP Dept. of Agriculture, Trade and Consumer Protection					
DH&SS Dept. of Health & Social Se Reminder: If the release is of an EHS or CERC	CLA chemical at or above the reportable quantity, the responsible party					
must also submit a follow-up written report per Fed. Statutes, as soon as practicable, to the State Emergency Response Board, Box 7865, Madison, WI 53707-7865, the Local Emergency Planning Committee and the MRC,						
release raisso	d pland tellison					
Signature of DEG Duty Officer	l Signature of Preparer					